UNITED STATES PATENT OFFICE.

HERBERT N. McCOY, OF CHICAGO, ILLINOIS, ASSIGNOR TO CARNOTITE REDUCTION COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF COLORADO.

ART OF RECOVERING RADIUM.

1,292,341. Specification of Letters Patent. Patented Jan. 21, 1919.

No Drawing. Application filed October 22, 1917. Serial No. 197,881.

To all whom it may concern:

Be it known that I, HERBERT N. McCOY, a citizen of the United States, residing at Chicago, in the county of Cook and State of Illinois, have invented a new and useful Improvement in the Art of Recovering Radium, of which the following is a specification.

My invention relates to the art of recovering radium and particularly to a treatment by which a radium concentrate may be prepared from a radium bearing mass of silicious minerals.

As is well known, radium occurs in very minute quantities in all uranium bearing minerals, the mineral known as carnotite being, however, by far the most important. Pure carnotite, a vanadate of potassium and uranium containing one three-millionth as much radium as uranium, is very rarely found massive, the so-called carnotite of commerce which is produced and extensively worked for the recovery of uranium containing usually but a small proportion (from 3 to 15 per cent.) of pure carnotite, the balance consisting of silica in the form of quartz sand and silicates, together with more or less earthy material.

Two general procedures for the recovery of radium from this so-called carnotite are at present in use. In the one, the radium along the barium, which usually accompanies it, is dissolved in nitric or hydrochloric acid, with or without previous preparatory treatment, leaving behind insoluble quartz sand and silicates. By this procedure (described in United States Bureau of Mines, Bulletin 104, pages 25 and 27) more or less of the uranium and vanadium pass into solution. According to the other procedure the carnotite is treated with sulfuric acid and afterward with water, in such a manner as to bring uranium and vanadium into solution as sulfates and to leave the radium and barium, whose sulfates are insoluble, in the undissolved residue. (See my prior United States Patent No. 1,098,282, granted May 26, 1914). After such treatment the undissolved portion contains in addition to the insoluble sulfates of the radium and barium large amounts of quartz sand and silicates. A large part of the coarser quartz sand may be separated by mechanical means, but the remaining radium bearing residue still contains a very considerable mass of fine quartz sand and silicates. The recovery of radium from this residue has heretofore been accomplished by the conversion of the silica and insoluble silicates into sodium silicate, by treatment with sodium carbonate or sodium hydroxid, the soluble sodium silicate being then dissolved in water, leaving an insoluble radium bearing concentrate largely freed of silica and silicates. This known procedure has many disadvantages, probably the most marked being that part of the radium goes into solution and is lost with the discarded sodium silicate solution.

I have discovered that it is possible to effect a better separation of silica and insoluble silicates from radium sulfate by the action of a water solution of hydrofluoric acid, whereby hydro-fluo-silicic acid is formed and dissolved by the water present, while the radium compound is left in the undissolved residue. The material, before treatment with hydrofluoric acid, contains more or less barium-sulfate, the presence of which is believed to aid in retaining the radium sulfate in the undissolved residue. In accordance with this theory, an ore which is low in barium is believed to be benefited by the addition of about ten pounds of barium-chlorid per ton. I prefer, also, to employ other mineral acids, such as sulfuric and hydrochloric, together with the hydrofluoric acid, these other acids dissolving bases such as aluminum, whose silicates are attacked by the hydrofluoric acid. The presence of sulfuric acid in the batch is also believed to be desirable as insuring the presence of the barium and radium as sulfates.

As illustrative of the procedure to be followed in carrying out my invention, the following example is given: One ton of commercial carnotite, which may, for example, consist of ten per cent. pure carnotite admixed with 90 per cent. sand and earthy material, is ground to 30-mesh and mixed with 600 pounds of concentrated sulfuric acid and 400 pounds of water. The mixture is dried at a temperature of 100° C. until the water has been largely driven off and is then baked at 250° C. The product of this treatment is a green solid containing the sulfates of all bases present, principally vanadium, uranium, aluminum, iron, magnesium, calcium and barium, together with the radium. This green solid is stirred with water, decanted, and the decanted liquor filtered. As the result of this treatment, the green solid